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Contents

Articles

Milan Melník, Marian Koman, Daniela Hudecová, Ján Moncol, Barbora Dudová, Tadeusz Glowiak, Jerzy Mroziński, Clive E. Holloway

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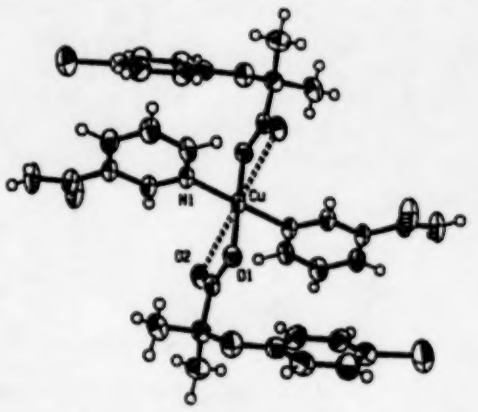
Spectral and magnetic properties and bio-activity of copper(II) clofibriates
Part I. Crystal and molecular structure of *trans*-Cu(clofibriate)$_2$(nicotinamide)$_2$

Cu(clofibriate)$_2$, Cu(clofibriate)-caffeine, and Cu(clofibriate)$_2$(LN)$_2$ (LN = nicotinamide or 3-pyridylcarbinol) were synthesized and characterized by elemental analysis, electronic and EPR spectra, and magnetic susceptibility (4.5–300 K). X-ray analysis results for Cu(clofibriate)$_2$(nicotinamide)$_2$ support a pseudo-octahedral arrangement about the Cu(II) atom for Cu(clof)$_2$, Cu(clof)$_2$(LN)$_2$ (spin $S = 1/2$). For Cu(clof)$_2$caf the dimeric (spin $S = 1$) structure is indicated.

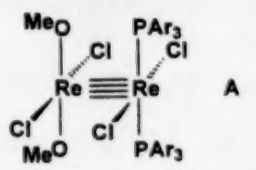


Sophia S. Lau, Phillip E. Fanwick, Richard A. Walton

Inorg. Chim. Acta 308 (2000) 8

The synthesis and characterization of multiply bonded dirhenium(III) alkoxide complexes containing two or three alkoxide ligands, including further studies on complexes of the type $Re_2Cl_4(OMe)_2(PAr_3)_2$ (Ar = aryl)

Reactions of the dirhenium(III) synthon *cis*-$Re_2(\mu\text{-}O_2CCH_3)_2Cl_4(H_2O)_2$ in refluxing methanol with triarylphosphines (PAr_3), which combine relatively low basicity with moderately large cone angle, produce alkoxides of composition $Re_2Cl_4(OMe)_2(PAr_3)_2$ (structure **A**) when PAr_3 = P(*p*-tolyl)$_3$, P(*m*-tolyl)$_3$, P(*p*-ClPh)$_3$, P(*p*-MeOPh)$_3$ and PPh_3. The first multiply bonded tris-alkoxide of dirhenium(III), $Re_2Cl_3(OEt)_3(PPh_3)_3$, has been prepared from the reactions of $Re_2Cl_6(PPh_3)_2$ and $Re_2Cl_4(OEt)_2(PPh_3)_2$ with NaOEt in ethanol.



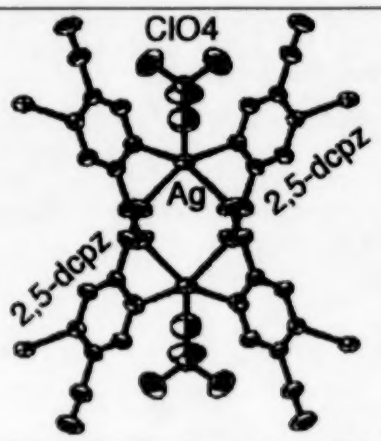
A

Yusaku Suenaga, Toru Kamiya, Takayoshi Kuroda-Sowa, Masahiko Maekawa, Megumu Munakata

Inorg. Chim. Acta 308 (2000) 17

Structural control of silver(I) coordination polymers: construction of two-and three-dimensional frameworks of silver(I) ions bridged by dicyanopyrazine derivatives

Two complexes [Ag(2,5-dcpz)(ClO_4)] and [Ag(2,5-dcpz)(CF_3SO_3)] have been prepared from different counter anions and structurally characterized (2,5-dcpz = pyrazine-2,5-dicarbonitrile). Both have four bridging ligands and one oxygen atom three dimensional structure with cavity. We could successfully control the dimensional structure of silver(I) complex comparing with [Ag(2,3-dcpz)(ClO_4)].

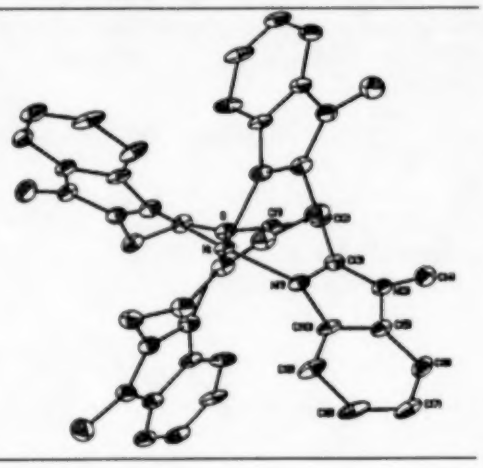


Jayakumar G. Gilbert, Anthony W. Addison, Raymond J. Butcher

Inorg. Chim. Acta 308 (2000) 22

Some nickel(II) complexes with pentadentate and tridentate heterocyclic N- and S-donor ligands

Nickel(II) complexes of three different ligands were synthesized. The complexes were characterized by X-ray crystallography, UV–visible, ESR spectrometry and electrochemical methods. The structures are either pseudooctahedral or tetragonally distorted.



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Smaïl Triki, Florence Bérézovsky, Jean Sala Pala, Maria-Teresa Garland

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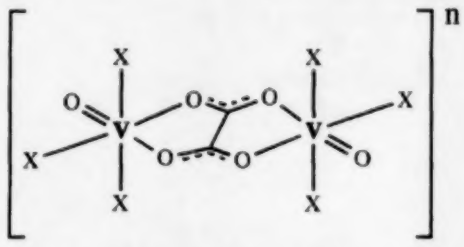
Synthesis and characterisation of new binuclear oxo-vanadium(IV) complexes with a bis(bidentate)oxalate bridge. Crystal structures of the cationic and anionic complexes $[V_2O_2(C_2O_4)(H_2O)_6]Cl_2{\cdot}4Bu_4NCl{\cdot}H_2O$ and $[FeCp_2^*]_2[Et_4N]_2[V_2O_2(C_2O_4)(NCS)_6]$

The new oxalate–vanadyl compounds $[V_2O_2(C_2O_4)(H_2O)_6]Cl_2{\cdot}4Bu_4NCl{\cdot}H_2O$ (**1**) and $[Et_4N]_4[V_2O_2(C_2O_4)(NCS)_6]$ (**2′**) have been synthesized starting from V_2O_5 and $VOSO_4{\cdot}5H_2O$ respectively. Metathesis of **2′** with $[FeCp_2^*]BF_4$ gave a partial substitution of the cations affording $[FeCp_2^*]_2[Et_4N]_2[V_2O_2(C_2O_4)(NCS)_6]$ (**2**). In **1** and **2** the centrosymmetric binuclear vanadium species present a bis(bidentate)oxalate ligand and an almost planar $[V_2O_2(C_2O_4)X_2]$ unit.



Linda Lettko, John S. Wood, Marvin D. Rausch

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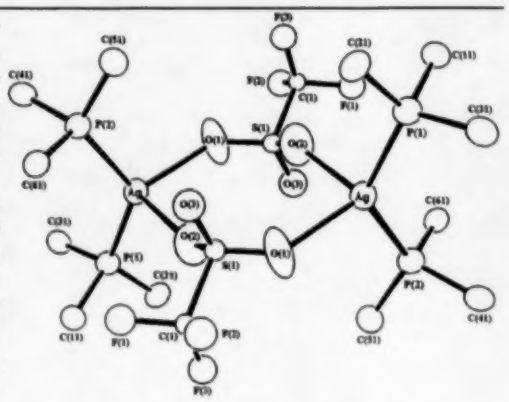
Synthesis of (phosphine)silver(I) trifluoromethanesulfonate complexes and the molecular structure of di-μ-trifluoromethylsulfonate-(tetrakis-triphenylphosphine)disilver(I)

Reactions of silver trifluoromethanesulfonate (triflate) and equimolar amounts of triphenylphosphine, 1,2-bis(diphenylphosphino)ethane, bis(diphenylphosphino)methane, or 1,1-bis(diphenylphosphino)-ethane in either DME or CH_2Cl_2 solution produced the corresponding 1:1 complexes $[(L)Ag(I)(SO_3CF_3)]$ (**2–5**) in >80% yields. A related 2:1 complex $[(Ph_3P)_2Ag(I)(SO_3CF_3)]$ (**1**) was also obtained using 2 equivalents of Ph_3Ph. Both ^{31}P NMR variable-temperature solution spectra as well as solid-state spectra were recorded for the new complexes, and were generally helpful in structural assignments. An X-ray crystallographic study of **1** shows this to be a dimeric complex with bridging triflate ions; the silver atoms having distorted tetrahedral coordination.

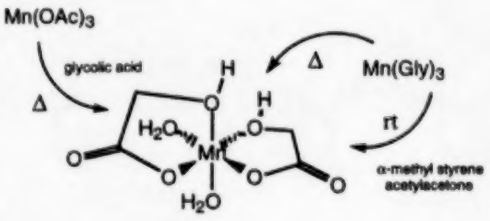


Gagik G. Melikyan, Fredrick Amiryan, Mandana Visi, Kenneth I. Hardcastle, Barney L. Bales, Gayane Aslanyan, Shaliko H. Badanyan

Inorg. Chim. Acta 308 (2000) 45

Manganese(II)/(III) glycolates: preparation, X-ray crystallographic study, and application in radical cycloaddition reactions

Novel methods for stereoselective synthesis of the chiral racemic $Mn(Gly)_2$ have been elaborated using high and low temperature transformations of $Mn(Gly)_3$ and $Mn(OAc)_3$. The X-ray crystal structure is temperature-independent, and it represents, by thermodynamic stability, the second most unstable isomer among five alternative structures. For the first time, $Mn(Gly)_3$ is used as a mediator for radical cycloadditions of β-dicarbonyl compounds to alkenes.

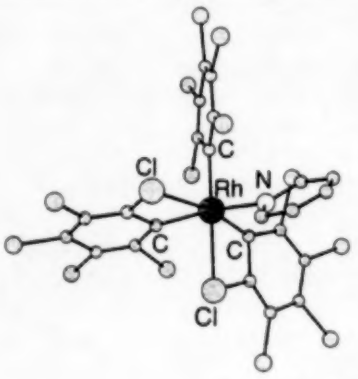


María P. García, Ana P. Martínez, M. Victoria Jiménez, Carmen Siurana, Luis A. Oro, Fernando J. Lahoz, Antonio Tiripicchio

Inorg. Chim. Acta 308 (2000) 51

Reactivity of rhodium(III) complexes containing chelating pentachlorophenyl ligands. Molecular structures of *fac*-$[Rh(C_6Cl_5)_3(py)]$ and *mer*-$[Rh(C_6Cl_5)_3(^tBuNC)_3]$

The reactivity of pentachlorophenyl rhodium(III) complexes containing Rh–*o*-Cl secondary bonds towards neutral N- or P-donor ligands is explored. The crystal structures of *fac*-$[Rh(C_6Cl_5)_3(py)]$ and *mer*-$[Rh(C_6Cl_5)_3(^tBuNC)_3$, with and without Rh–*o*-Cl interactions, are also presented.

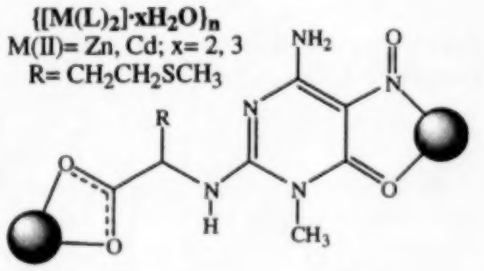


R. López-Garzón, P. Arranz-Mascarós, M.L. Godino-Salido, M.D. Gutiérrez-Valero, A. Pérez-Cadenas, J. Cobo-Domingo, J.M. Moreno

Inorg. Chim. Acta 308 (2000) 59

Solution study and 2-D layered structures of zinc(II) and cadmium(II) complexes with *N*-2-(6-amino-3,4-dihydro-3-methyl-5-nitroso-4-oxo-pyrimidinyl)-L-methionine as ligand

The Zn(II) and Cd(II) complexes obtained with HL have been studied in solution, $[ZnL]^+$, $[ZnL]_2$ and $[Cd(HL)_2]^{2+}$, and in solid state, $\{[M(L)_2]{\cdot}xH_2O\}_n$. The later consist of layered structures very similar even though they are obtained from different mononuclear complexes detected in solution.



Effendy, Giancarlo Gioia Lobbia, Claudio Pettinari, Carlo Santini, Brian W. Skelton, Allan H. White

Inorg. Chim. Acta 308 (2000) 65

Synthesis, characterization and X-ray structural studies of novel dinuclear silver(I) complexes of poly(azolyl)borate ligands

A series of Ag(I) complexes of tris- and tetrakis-(pyrazolyl)hydroborates, tetrakis(imidazol-1-yl)borate, and hydrotris(3-methyl-1-imidazolyl-2-thione)borate, have been synthesized and characterized through analytical and spectral measurements. A single crystal study shows $[Ag(Tm)]_2 \cdot 2CHCl_3$ to be binuclear, the core of the dimer being a four-membered $Ag(\mu\text{-}S)_2Ag$ ring.

Pu-Hui Xie, Yuan-Jun Hou, Tian-Xin Wei, Bao-Wen Zhang, Yi Cao, Chun-Hui Huang

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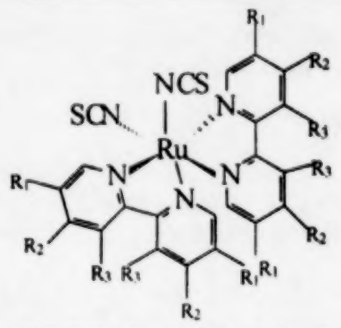
Synthesis and photoelectric studies of Ru(II) polypyridyl sensitizers

The photoelectrochemical properties of the three dyes adsorbed on the surface of the nanocrystalline TiO_2 electrodes as well as on self-assembly monolayers (SAMs) were investigated.



1 *cis*-Ru(3,3'-dcbpy)$_2$(NCS)$_2$ R_1=R_2=H, R_3=COOH
2 *cis*-Ru(4,4'-dcbpy)$_2$(NCS)$_2$ R_1=R_3=H, R_2=COOH
3 *cis*-Ru(5,5'-dcbpy)$_2$(NCS)$_2$ R_2=R_3=H, R_1=COOH

Xiaoyuan Chen, Frank J. Femia, John W. Babich, Jon Zubieta

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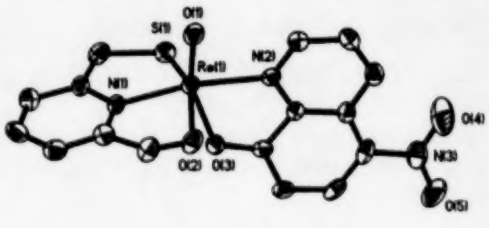
The syntheses and structures of '3 + 2' and '2 + 2 + 1' oxorhenium mixed-ligand complexes employing 8-hydroxy-5-nitroquinoline as the bidentate N,O donor ligand

One-pot reaction of $[ReOCl_3(PPh_3)_2]$ with the bidentate N,O donor ligand 8-hydroxy-5-nitroquinoline (HL) and dianionic tridentate ligands H_2L^n (where $H_2L^1 = HOC_6H_4\text{-}2\text{-}CH{=}NC_6H_4\text{-}2\text{-}OH$; $H_2L^2 = HOC_6H_4\text{-}2\text{-}CH{=}N\text{–}C_6H_4\text{-}2\text{-}SH$; $H_2L^3 = HOC_6H_4\text{-}2\text{-}CH{=}NN{=}C(NHC_6H_5)\text{–}SH$; $H_2L^4 = 2\text{-}CH_2OH\text{–}C_5H_3N\text{-}6\text{-}CH_2SH$; and $H_2L^5 = 2\text{-}CO_2H\text{–}C_5H_3N\text{-}6\text{-}CO_2H$) in the presence of triethylamine afforded a series of '3 + 2' complexes $[ReO(L^n)(L)]$ **2**–**6** with distorted octahedral geometry, featuring the phenolic oxygen trans to the Re=O group. In contrast, the potentially tridentate ligand 2,6-pyridinedimethanol (H_2L^6) acted as a bidentate N,O donor ligand to form a '2 + 2 + 1' oxorhenium complex $[ReO(OCH_3)(HL^6)(L)]$ **7**. Furthermore, cleavage of the C=N double bond in the preparation of **3** was encountered and a side product $[ReO(PPh_3)(\eta^2\text{-}NHC_6H_4\text{-}2\text{-}S)(L)]$ **8** was isolated. When H_2L^5 was employed, reduction of HL to 8-hydroxy-5-aminoquinoline was realized, and a new '2 + 2 + 1' complex $[ReO(OCH_3)(\eta^2\text{-}O\text{-}5\text{-}NH_2\text{–}C_9H_5N)(L)]$ **9** along with **6** was obtained.

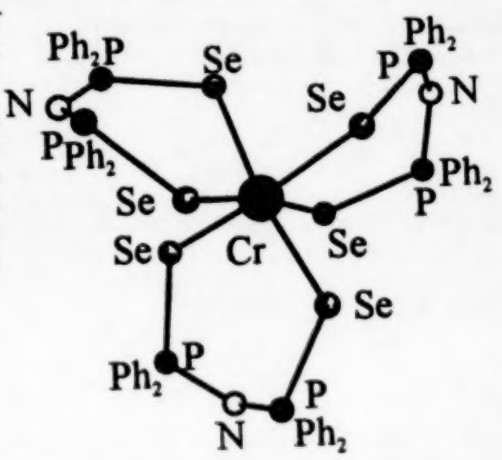


Virginie Béreau, Perumal Sekar, Craig C. McLauchlan, James A. Ibers

Inorg. Chim. Acta 308 (2000) 91

Syntheses and characterization of imidodiphosphinoselenido complexes of V(IV), V(III), and Cr(III): $[VO\{N(SePPh_2)_2\}_2]\cdot CH_2Cl_2$, $[V\{N(SePPh_2)_2\}_3]\cdot CH_2Cl_2$, and $[Cr\{N(SePPh_2)_2\}_3]\cdot CH_2Cl_2$

The imidodiphosphinoselenido complexes of V(IV), V(III), and Cr(III) — $[VO\{N(SePPh_2)_2\}_2]\cdot CH_2Cl_2$ (**1**), $[V\{N(SePPh_2)_2\}_3]\cdot CH_2Cl_2$ (**2**), and $[Cr\{N(SePPh_2)_2\}_3]\cdot CH_2Cl_2$ (**3**) — have been prepared from the reaction of $VCl_3(THF)_3$ (**1** and **2**) or $CrCl_2$ (**3**) with $K[N(SePPh_2)_2]$ in CH_2Cl_2. In each of these compounds the $[N(SePPh_2)_2]^-$ ligand exhibits an *Se,Se'*-bonding chelation to the metal center.

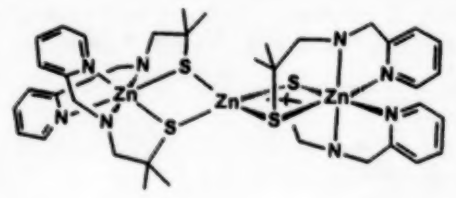


U. Brand, H. Vahrenkamp

Inorg. Chim. Acta 308 (2000) 97

Zinc complexes of the new *N,N,S* ligand *N*-(2-mercaptoisobutyl)(picolyl)amine

The title ligand MBPAH forms zinc complexes with three different coordination modes: tetrahedral ZnN_2S_2 in $(MBPA)_2Zn$, octahedral ZnN_4S_2 and tetrahedral ZnS_4 in $[(MBPA)_4Zn_3]X_2$, and trigonal bipyramidal ZnN_2S_2O in dimeric (MBPA)Zn-acetate.

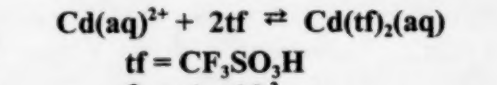


Wei Xu, Ronald F. Evilia

Inorg. Chim. Acta 308 (2000) 103

NMR study of cadmium(II)–triflate coordination

The NMR chemical shift of aqueous cadmium triflate solution is found to vary with concentration. The chemical shift/concentration variation is analyzed to yield a value of 4×10^{-3} for the overall formation constant of the bis complex (β_2). The chemical shift of $Cd(tf)_2$ is found to be -180 ppm from aqueous Cd^{2+}.

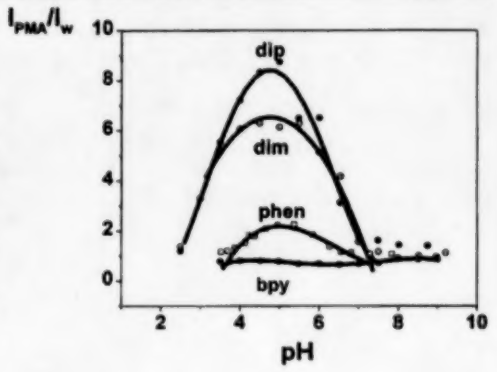


Silvia Ronco, Brian Persing, Roy Mortinsen, Jackie Barber, Sabrina Shotwell

Inorg. Chim. Acta 308 (2000) 107

Probing the presence of hydrophobic sites in anionic polyelectrolytes with tris(polypyridine)-chromium(III) complexes

The emission properties of tris(polypyridine)-chromium(III) complexes in aqueous solutions are dramatically modified in the presence of anionic polyelectrolytes. The largest effects were observed for tris(4,7-diphenylphenanthroline)chromium(III) bound to poly(methacrylic acid), where the emission intensity was enhanced 25.66 times with respect to pure water. Potential use of these complexes to detect hydrophobic sites is discussed as a function of experimental parameters.



Rolfe H. Herber, Otto J. Scherer

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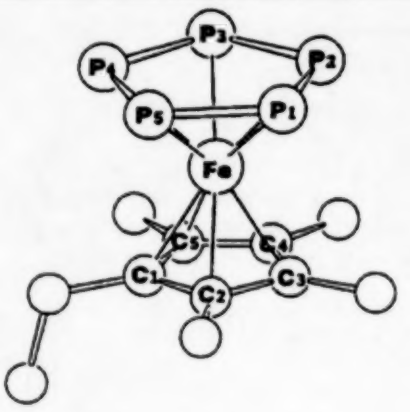
Bonding, metal atom motion, and evidence for a phase transition in Fe metallocenes with a *cyclo*-P_5 ligand

The electron density and charge symmetry around the metal atom in η^5-Cp*Fe-η^5-P_5, and the kinetics of the metal atom motion, have been studied over the range $85 \leq T \leq 380$ K by temperature-dependent ^{57}Fe Mössbauer spectroscopy, and the results have been compared to analogous iron metal-organic complexes. Evidence for a phase transition is presented.

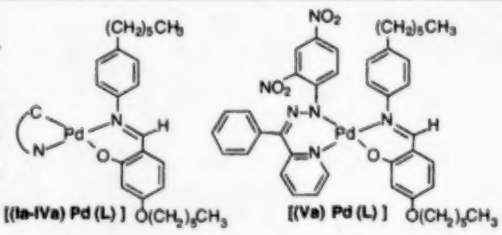


Iolinda Aiello, Alessandra Crispini, Mauro Ghedini, Massimo La Deda, Francesco Barigelletti

Inorg. Chim. Acta 308 (2000) 121

Synthesis and characterization of a homologous series of mononuclear palladium complexes containing different cyclometalated ligands

The synthesis, characterization and spectroscopic properties of the homologous series of mononuclear cyclometalated complexes [(Ia–Va)Pd(L)] derived from **HL** [2-hydroxy-4-(*n*-hexyloxybenzylidene)-4′-*n*-hexylaniline] and the deprotonated form of **I**–**V** (**I** = 2-phenylpyridine; **II** = benzo[h]quinoline; **III** = azobenzene; **IV** = 2-benzoylpyridine; **V** = phenyl-2-pyridylketone-2,4-dinitrophenylhydrazone) are described. The molecular structures of [(IVa)Pd(L)] and [(Va)Pd(L) have been determined by single-crystal X-ray analysis. Compounds [(IIIa)Pd(L)] and [(Va)Pd(L)] show solvatochromism.

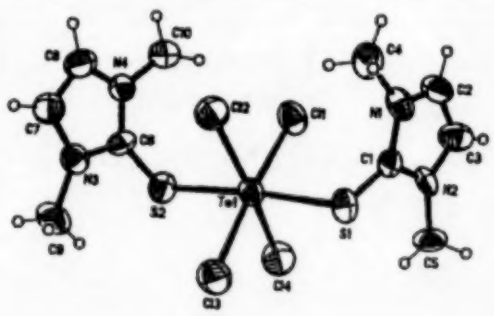


Daniel J. Williams, Vicky L.H. Bevilacqua, Peter A. Morson, William T. Pennington, George L. Schimek, Nancy T. Kawai

Inorg. Chim. Acta 308 (2000) 129

Main group metal halide complexes with sterically hindered thioureas
Part XVII. The crystal and molecular structures of two new tellurium chloride complexes with 1,3-dimethyl-2(3*H*)-imidazolethione

Two new complexes of tellurium chloride with 1,3-dimethyl-2(3*H*)-imidazolethione (dmit) are reported. The new compounds were prepared out of boiling acetonitrile from a previously reported compound, $TeCl_2(dmit)_4$. X-ray crystallographic studies as well as spectroscopic data reveal a new square planar Te(II) chloro complex and a new distorted octahedral Te(IV) chloro complex with dmit.



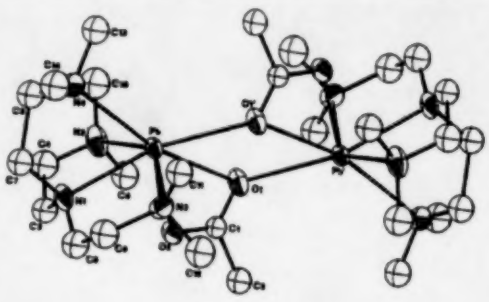
Notes

Franco Cecconi, Carlo A. Ghilardi, Stefano Midollini, Annabella Orlandini

Inorg. Chim. Acta 308 (2000) 135

Lone pair activity in lead(II) compounds. X-ray structure of the dimeric complex $[(trenMe_6)Pb(O_2CCH_3)]_2(BPh_4)_2$, $trenMe_6 = N(CH_2CH_2N(CH_3)_2)_3$

The compound $[(trenMe_6)Pb(O_2CCH_3)]_2(BPh_4)_2$, $trenMe_6 = N(CH_2CH_2N(CH_3)_2)_3$, has been prepared and structurally characterized. The complex cation displays a dimeric structure where each lead metal is linked by one $trenMe_6$ ligand and by three oxygens of two bridging acetate groups in a hepta-coordination. The angular distribution of the ligands around the metal as well as the values of the Pb–L distances, are indicative of the presence of a stereochemically active lone pair on Pb(II).

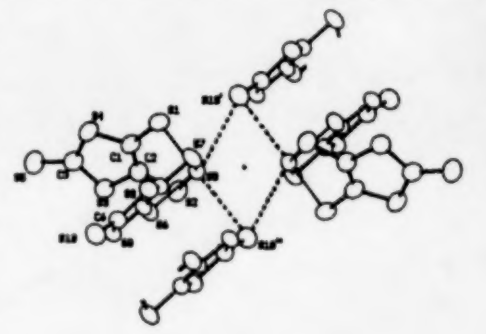


P. Ganis, D. Marton, G.M. Spencer, J.L. Wardell, Solange M.S.V. Wardell

Inorg. Chim. Acta 308 (2000) 139

The molecular and crystal structure of tetraphenylphosphonium antimony(III) bis-dmit. A comparison with similar complexes

The molecular and crystal structure of tetraphenylphosphonium antimony(III) bis-dmit (H_2-dmit = 4,5-dimercapto-1,3-dithiole-2-thione) has been synthesized and structurally characterized. The coordination about Sb is almost undistorted pentagonal bipyramidal and is achieved through intraanionic and secondary interanionic bond interactions between Sb and thiocarbonyl sulfur atoms.The crystal structure is characterized by a linear polymeric association along *c*.

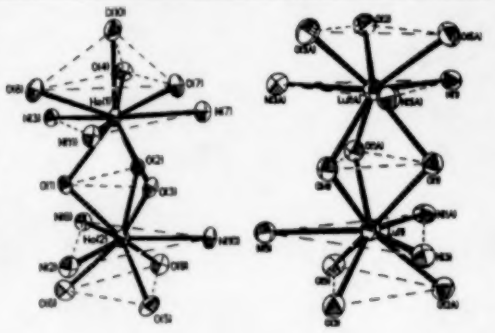


Xian-He Bu, Miao Du, Lei Zhang, Xu-Bo Song, Ruo-Hua Zhang, Thomas Clifford

Inorg. Chim. Acta 308 (2000) 143

Novel homobinuclear lanthanide(III) complexes with isonicotinoyl hydrazone: synthesis and coordination chemistry

Two novel homobinuclear Ln(III) complexes with isonicotinoyl hydrazone (**L**) have been synthesized and characterized. The two Ho(III) ions in **I** have different coordination environments with nine and ten coordination numbers, while in **II**, both Lu(III) ions are nine-coordinate. **L** acts as five-dentate ligand with keto-form coordination, and the Ln(III) ions are bridged by three phenolic oxygen atoms.

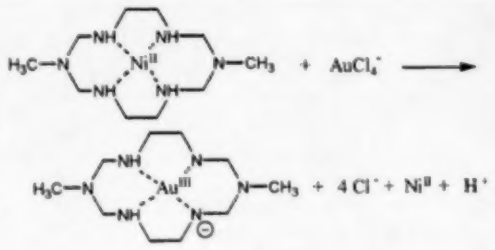


Hyochoon Bang, Eun Ja Lee, Eun Young Lee, Junghun Suh, Myunghyun Paik Suh

Inorg. Chim. Acta 308 (2000) 150

Transmetallation of nickel(II) ions in a nickel(II) azamacrocyclic complex with gold(III) ions

The Ni(II) macrocyclic complex $[Ni(L)]^{2+}$ reacts with $HAuCl_4$ in MeCN, and the Ni(II) ion in the macrocyclic complex is exchanged with the Au(III) ion. The kinetic study on the transmetallation reaction shows that the rate is inhibited by acid and Cl^-, which is explained in mechanistic terms.



Thérèse A. Coffey, Graeme Hogarth, Simon P. Redmond

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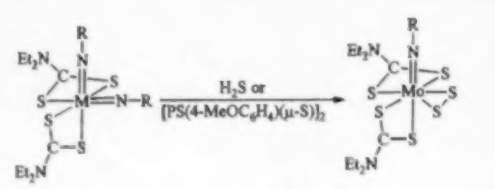
Replacement of imido by disulfido at the molybdenum(VI) centre: reactivity of bis(imido) complexes $[Mo(\eta^2\text{-}S_2CNEt_2)_2(NR)_2]$ with hydrogen sulfide and Lawesson's reagent: X-ray crystal structure of $[Mo(\eta^2\text{-}S_2CNEt_2)_2(\eta^2\text{-}S_2)(N\text{-}2,6\text{-}Cl_2C_6H_3)]$

Reactions of $[Mo(\eta^2\text{-}S_2CNEt_2)_2(NR)_2]$ with hydrogen sulfide and Lawesson's reagent results in formation of imido–disulfido complexes $[Mo(\eta^2\text{-}S_2CNEt_2)_2(\eta^2\text{-}S_2)(NR)]$ as a result of imido–disulfido exchange and $[Mo(\eta^2\text{-}S_2CNEt_2)_2(\eta^2\text{-}S_2)(N\text{-}2,6\text{-}Cl_2C_6H_3)]$ has been characterised crystallographically.



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